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THE SUSCEPTABILITY OF MATERIALS TO HYDROGEN EMBRITTLEMENT FROM CHEMICAL MILLING OPERATIONS

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ABSTRACT

Several steel and titanium alloys were chemically milled in acid baths generally similar to those employed commercially. The steels; AISI 4340, 431 stainless, 301 FH stainless, A286 stainless, AM350, and AM355CRT, were chemically milled in a hydrochloric - nitric - phosphoric acid formulation. The titanium alloys, Ti-5Al-2.5Sn, Ti-6AL-4V, and Ti-13V-11Cr-3Al, were chemically milled in a hydrofluoric acid bath.

The chemically milled materials were investigated for susceptability to hydrogen embrittlement by use of a constant strain rate bend tester designed and built at Convair-Astronautics. This test technique has proven to be especially suitable for the investigation of hydrogen embrittlement of sheet materials. The bend test proved quite sensitive and is inexpensive, rapid and easy to perform.

Of the steels investigated, only the 4340 and 301 cold worked stainless were hydrogen embrittled by the chemical milling process. Although most of the other steels were similar in structure and of equal or greater tensile strength, they were not embrittled by hydrogen pickup from chemical milling as evaluated by the constant strain rate bend test.

The alpha titanium alloy Ti-5Al-2.5Sn was not embrittled by hydrogen introduced by chemical milling and the alpha-beta alloy Ti-6Al-4V showed only minor embrittlement. However, the beta alloy Ti-13V-11Cr-3Al was severely embrittled by chemical milling.

It was found that the original ductility of the hydrogen embrittled materials could be restored by appropriate recovery treatments. The ease and rapidity with which ductility was restored was found to depend upon the material. For example, hydrogen embrittled 4340 steel was found to be free from hydrogen embrittlement after holding between 8 and 33 hours at room temperature.

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INTRODUCTION

One of the phenomena encountered in steels heat treated to relatively high strength levels is the occurrence of brittle delayed failures in service. For example, electroplated high strength steel bolts have shown delayed failures at the root of the threads or in the head fillet; similarly, other electroplated high strength steel parts have been known to fail in a brittle fashion under the influence of static loads. This has been observed under relatively mild service conditions in spite of precautions taken to minimize or eliminate the commonly known embrittling factors such as poor fillet design and improper thermal treatments. Service failures have been reported in plain carbon steel springs heat treated to high strength levels and subsequently plated or pickled and in alloy steels aircraft landing gear components heat treated to high strength and then plated with cadmium.

In view of these observations, it appeared that the phenomenon of delayed failure under static load was associated with first, steels heat treated to high strength levels, and second, exposure to a hydrogen environment in processing. Since the recognition of hydrogen embrittlement of steels, considerable evidence has been obtained to show that this phenomenon occurs in many other materials.

It is now well established that under certain conditions hydrogen can have an adverse effect on the mechanical properties of steels, particularly high strength steels, some titanium alloys, and to a lesser extent some other ferrous and non-ferrous alloys. The chief effects are a marked decrease in ductility on slow straining and a propensity toward delayed failures under static loads even at stresses far below the yield strength. In general, impact resistance and fatigue properties remain unaffected. The Young's modulus is not changed and the shape of the true stress-strain curve remains the same. The ultimate tensile strength of notched and unnotched specimens are affected only slightly.

REVIEW OF LITERATURE

Hydrogen Embrittlement of Steels

The following is a brief review of some of the many observations of the effects of hydrogen. An exhaustive literature survey has been made by Buzzard and Cleaves (1) and other work includes summaries of previous information together with bibliographies (2,3,4,5,6,7 and 8).

Hydrogen may be introduced into steel from a variety of sources. The steel-making process itself is one major origin of hydrogen (9,10) and it is particularly troublesome in large section sizes (11,12). Other processing operations such as acid pickling (13,14) and electroplating (15,16,17) are recognized sources which have been studied. It is quite probable that the chemical milling process will introduce hydrogen.

One interesting aspect of hydrogen in steel is the effect of cold rolling. Darken and Smith (18) showed that the maximum amount of hydrogen absorbed in pickling increased linearly by increasing the degree of cold working prior to pickling. They also found that the rate of absorption showed a similar dependence on the degree of deformation. Bend tests have shown that cold worked steel is more susceptible to embrittlement than annealed steel (19).

The strong dependence of hydrogen embrittlement on strain rate and testing temperature has been verified by a number of investigations (20,21,22,23,24). For example, it is well known that hydrogen charged specimens which exhibit embrittlement at moderate strain rates and temperatures can be made to show normal ductilities when tested at high strain rates or at low temperatures.

The strength level to which steel is heat treated also has a strong effect on the susceptibility to hydrogen embrittlement. In general, higher strength levels increase the susceptibility of a given steel to hydrogen embrittlement (25,26,27,28).

Numerous studies have been made in which recovery from the embrittled state has been observed to occur with elapsed time at room or somewhat elevated temperatures (8,16,17,19,23). Various criteria, such as bend tests, cup tests elongation, reduction of area and notched rupture tests have been used as an index of embrittlement. In general, the degree of embrittlement decreased with increased holding time and temperature and complete recovery of ductility was eventually attained if the charging conditions were not too severe. On the other hand, when the strength level was high and the charging conditions severe (high hydrogen contents), baking treatments which were effective in producing recovery at lower strength levels were not able to produce complete recovery at the higher strength levels.

In review, then, it has been established that the degree of embrittlement of steels is influenced by:

(1) hydrogen content
(2) ultimate tensile strength
(3) temperature of test
(4) time elapsed after charging with hydrogen
(5) rate of application and magnitude of stress
(6) temperature and time of hold of hydrogen relief treatment

Hydrogen Embrittlement of Titanium

Hydrogen embrittlement in titanium and its alloys manifests itself in either high or low strain rate embrittlement and thus differs in this respect from hydrogen embrittlement of steels. In general, the low strain rate embrittlement of titanium is quite similar to steels and depends on hydrogen content, ultimate tensile strength, temperature and rate of application and magnitude of stress (24,28,29). Low strain rate embrittlement has been shown to occur in all-alpha and all-beta alloys as well as alpha-beta alloys (28,29).

Unalloyed titanium has been reported to be most sensitive to hydrogen embrittlement at high strain rates (impact speeds) (30). Since the solubility of hydrogen in unalloyed titanium is quite low (approximately 30 ppm), titanium hydride (TiH) can be readily formed. This hydride is reported to have a face centered cubic or a face centered tetrogonal structure and is thought to be responsible for the high strain rate embrittlement. Alpha stabilizing additions, such as aluminum, seem to increase the solubility of hydrogen in titanium and, consequently, lower the sensitivity of the alloy to hydride induced impact embrittlement (31).

Although the effects of hydrogen in titanium is in many ways similar to that in steels, there are several major differences between the steels and alpha-beta alloys. The amount of hydrogen required to embrittle titanium alloys is an order of magnitude greater than steel. Steels will outgas at room temperature, titanium alloys will not. Titanium forms a hydride, steel does not. Also, some titanium alloys creep a significant amount at room temperature while little or no creep is found in steels at this temperature. For example, Troiano (29) has found creep failures in the Ti-5Al-2.5Sn alloy at high stresses and low hydrogen levels at room temperature. Some steels and titanium alloys have been found to be subject to strain aging and its associated effects on mechanical properties. However, it appears that hydrogen can cause strain aging in titanium while this is not true for steels. Low strain rate hydrogen embrittlement of alpha-beta titanium alloys has been called a strain aging phenomenon by Burte (32) and by Troiano (29).

Hydrogen Embrittlement of Other Materials

Hydrogen has been shown to have an embrittling effect in other materials such as nickel, vanadium, zirconium, columbium and several austenitic nickel chromium-iron alloys (29,33,34). However, in comparison to steel and titanium alloys, a very high hydrogen concentration is required to produce embrittlement. Although very little work has been down on other materials, enough has been established to show that, although an important factor, the crystal structure of an alloy does not provide an absolute criterion for predicting an alloy's susceptibility to hydrogen embrittlement.

For example, though austenitic iron-chromium-nickel alloys (face centered cubic structure) are in general less capable of being embrittled than ferritic (body centered cubic structure) and martensitic steels (tetragonal structure), they are definetly susceptible to hydrogen embrittlement if sufficient hydrogen is present. This susceptibility is a function of composition among other things.

An interesting illustration of the effect of composition has been provided by Troiano and Blanchard (29). They investigated the effects of adding chromium and iron to nickel and covered the range from commercially pure nickel to 25-20 stainless steel (all austenitic structures). It was found that the hydrogen embrittlement of the nickel-iron alloy decreased with increasing iron; an apparent anomaly since iron is the easiest metal to embrittle with hydrogen. These authors have attempted to explain this behavior by relating the decreased susceptibility to hydrogen embrittlement with increased iron content to the filling of the incomplete 3d bands of the transition metals.

Theories of Hydrogen Embrittlement

The theories to be discussed below are primarily aimed at explaining the slow strain rate hydrogen embrittlement of steels, although they may possibly apply to other materials.

An acceptable theory must explain the unusual characteristics of the embrittlement phenomenon. First, hydrogen embrittlement disappears at low and high test temperatures and is accordingly most severe in an intermediate temperature range. Second, in striking contrast to "conventional" embrittlement phenomena, hydrogen embrittlement is inversly dependent upon strain rate, i.e., it decreases with increasing strain rate.

No universally accepted theory of hydrogen embrittlement has been developed as yet. The current theories employed to explain hydrogen embrittlement can be grouped in two categories. One explains embrittlement on the basis of the pressure exerted by molecular hydrogen in lattice voids or defects, while the other attributes the embrittlement to the influence of hydrogen in the lattice.

Historically, the planar pressure theory advanced by Zapffe and co-workers was first used to explain the experimental observations (35). Other workers, Bastien and Azou (36), de Kazinczy (37), and Petch and Stables (38), have proposed mechanisms of hydrogen embrittlement which are, broadly speaking, variations of the planar pressure theory. These theories exhibit similar features, although the proposed mechanisms differ in detail. All investigators postulate the existance of a lattice containing defects. Depending on the author, these defects may be termed Griffith cracks, voids or lattice rifts. The implicit assumption is made that the voids are of large size when compared to the volume of the lattice unit cell. In all theories the source of damaging hydrogen is the hydrogen in solution which diffuses into and is localized in the voids or at the void surface. The specific details of the embrittlement mechanism depend upon whether the hydrogen at the void surface or in the void is assumed to be embrittling. Zapffe, Bastien and Azou and de Kazinczy assume that hydrogen in voids is damaging, whereas Petch and Stables believe that embrittlement is caused by hydrogen adsorbed on the void surface.

Each of the embrittlement mechanisms discussed above can be used to explain a great deal (but not all) of the observed experimental observations. Objections have been raised to the various theories based on certain aspects of the proposed mechanism. For example, Toh and Baldwin have raised an objection to all the above theories (39). They point out that in each case it is assumed that the distribution of total hydrogen (i.e. the hydrogen in solution and in the voids) is uniform throughout the steel. Whereas, in fact, experimental evidence indicates that frequently the hydrogen is confined to a thin layer near the surface of the steel. Troiano, et. al (40) critically evaluated these theories of hydrogen embrittlement by investigating the influence of plastic strain on the aging characteristics of hydrogenated steel. They found that the recovery curve was strongly modified by plastic deformation either prior or subsequent to hydrogenation and noted that the planar pressure theory was incapable of explaining the observed results. Furthermore, this work definely indicated that hydrogen in voids was really non-embrittling.

Based on these results, Troiano proposed a new theory to explain hydrogen embrittlement. The new theory adequately explains all observed effects of hydrogen including those found in recent studies. According to Troiano a critical combination of triaxial stress state and hydrogen concentration is necessary for initiation and subsequent propagation of a crack. Thus hydrogen controls crack propagation by diffusing ahead of (or to) the advancing crack. It should be noted that in this theory it is the hydrogen in solid solution in the lattice that is the embrittling agent whereas it is molecular hydrogen in the voids in the planar pressure theories.

Using Troiano's theory, it is possible to predict the crack kenetics of delayed failure due to hydrogen. When hydrogen concentration in a region of maximum triaxiality (notch, inclusion, microcrack, etc.) reaches a critical value a crack is nucleated. This initial crack should propagate instantaneously until stopped by the higher fracture stress of the adjacent material of lower hydrogen content. The continuation of cracking must then await the further diffusion of hydrogen to the new region of triaxiality near the base of the crack where another initiation will occur. Thus delayed failure of hydrogenated steel should proceed by a process of discontinuous crack initiation and growth. In a more recent work Troiano has shown that delayed failure in hydrogenated steel is, indeed, a process of discontinuous crack initiation and growth (41). Thus, considerable evidence has been found to support Troiano's theory of hydrogen embrittlement.

Hydrogen Embrittlement Test Techniques

In the past, many test techniques have been used to evaluate the hydrogen embrittlement susceptibility of many materials. A list of many of the tests employed divided into short duration and long duration tests is given below:

A. Short Duration

Test	Measure of embrittlement
1. Tensile	reduction of area
2. Bend over mandrel	angle of bend
3. Notched tensile	Notched tensile strength

B. Long Duration

Test	Measure of embrittlement
1. Stressed ring	time to failure
2. Stressed notched "C" ring	time to failure
3. Torqued bolt	time to failure
4. Sustained load bend	time to failure
5. Sustained load notched tensile	time to failure

In general, these tests have been employed throughout the aircraft industry as a go-no go quality control test of heat treated and electro-plated steels. Most of these tests, however, are not adequate for a quantitative evaluation of the phenomenon, and in some instances they are not even qualitatively adequate. This is because the quantities measured in these tests involve many other physical factors of complex nature which tend to cloud the significance of the results. Perhaps the regular tensile test is most typical in this respect (42). That all test techniques do not possess adequate sensitivity in detecting hydrogen embrittlement was shown in a recent project carried out by an ARTC project, W-95 (43). Most of the test techniques shown above were compared by using the same heat of steel embrittled to the same extent. Of all test techniques investigated, the sustained load notched tensile test (rupture test) was the most sensitive and reproducible.

One interesting feature of the sustained load notched tensile test (rupture test) is that by conducting tests at enough stress levels the lower critical stress (stress below which failure does not occur) can be determined with a notch of a given geometry (41). It appears, therefore, that data of some quantitative significance can be obtained by this test technique. However, the lower critical stress is also a function of notch geometry and it is difficult, if not impossible, to apply this data to actual design.

From a practical standpoint the notched tensile test may be too sensitive in many cases. The degree of embrittlement that can be tolerated in a given article is determined not only by the criticality of the article but also by the nature of the service stresses which are difficult to calculate rigorously. Thus complete freedom from embrittlement in a maximum sensitivity test is not at all necessary for many applications. For example, baking treatments which relieved failure of electroplated lockwashers were not sufficient to prevent delayed failure in notched tensile tests (17).

In general, the more sensitive tests are considerably more expensive and time consuming. Furthermore, most of the tests used at present are not suited to the testing of sheet materials. Therefore, it appears justifiable to examine other test methods to determine their suitability for testing sheet materials and their suitability as a possible replacement for the more expensive, long time test methods presently in use.

The ideal test technique should be inexpensive, short in duration, amenable to sheet material and sensitive to detection of hydrogen embrittlement at fairly low levels of embrittlement. Bend tests seem to satisfy all the above requirements but the last. Bend tests, as they have been carried out in the past, have proven so insensitive that their use seems highly undesirable. However, a new approach to bend testing was initiated a few years ago by Sacks (44). It is ideally suited to sheet material since it uses bending of thin strips. Bending of the sample is performed as a free end-loaded column at various constant strain rates. This test permits accurate measurement of the fracture strains of a steel over a wide range of embrittlement conditions. This test is further characterized by a nearly linear increase in strain with reduction in column height and is, therefore, a constant strain rate test (44,45). This is in contrast to most bend tests which are constant bend radius tests.

Therefore, this test technique was adopted for the present study of hydrogen embrittlement of sheet materials as effected by chemical milling.

EXPERIMENTAL PROCEDURES

Materials

Materials were selected on the basis of their present or potential use in the aircraft and missile industry in applications where chemical milling might be used as a processing tool. Nine materials were investigated; 4340 steel, 431 martensitic stainless steel, AM 350 semi-austenitic precipitation hardenable stainless steel, 301 and AM 355 stainless steels cold worked to high strength, A286 austenitic precipitation hardenable stainless steel and three titanium alloys - the all alpha alloy Ti-5Al-2.5Sn the alpha plus beta alloy Ti-6Al-4V and the all beta alloy Ti-13V-11Cr-3Al.

The chemical composition of the alloys are given in Table I. Chemical compositions were available or determined for several of the alloys but for others, particularly the titanium alloys, actual composition was not determined. Thus, only the normal composition range is reported in Table 1.

The sheet materials were sheared oversize and ground to final size to eliminate edge effects from shearing. Sample configuration and demensions are shown in figure 1. In all cases samples were machined to final size before heat treatment.

Heat Treatment

Three of the nine materials were investigated in the as-received condition. The all alpha titanium alloy, was investigated in the annealed condition. The 301 and AM 355 alloys were used "as-received". These alloys were cold rolled to high strength levels, AM 355 being the strongest material studied in this program.

All other materials were heat treated to strength by appropriate thermal treatments. Hardness measurements were made on all materials and tensile ultimate and tensile yield strengths were determined for many of the alloys.

The thermal treatments employed and the resulting mechanical properties are given in Table 2. Where actual tensile properties were not determined they were either estimated from hardness measurements or guaranteed minimum values have been reported. These values are so indicated in the Table.

Chemical Milling

Chemical milling was carried out in the laboratory using baths generally similar to those employed commercially for the various materials. All steels were chemically milled in the bath shown below at a temperature of 140°F.

Steel chemical milling bath

HCl	-	15% by volume
HNO_3	-	17%
H_3PO_4	-	31%
H_2O	-	37%
Fe	-	4 grams/liter

At this temperature the bath mills at a rate of about 1 mil/minute. The milling rate changed with the age of the bath and type of material, but, in general, the change was small.

The titanium alloys were chemically milled in the solution shown below at a temperature of 140°F. Milling rate was similar to that of the solution used for steel. However, the solution degenerated in milling power more rapidly than the steel solution.

Titanium Chemical Milling Bath

HF	- 23% by volume
H_2O	77% by volume
Chromic Acid	125 grams/liter

In both cases the milling bath was replaced with a new solution when the milling rate had decreased by a factor of 10 percent.

Except for the all beta titanium alloy, Ti-13V-11Cr-3Al, the bend samples were chemically milled from one side only. This was accomplished by masking one side and the edges with pressure sensitive lead tape. The chemical milled surface was then made the tension side during the bend test.

The all beta titanium alloy was milled from both sides; only the edges were taped. This was necessary since milling from one side only permitted the uneven release of internal stresses which caused the bend sample to deform severely.

The commercial proprietory baths differ slightly from the solutions used in this investigation. Generally, small additions are made to the commercial baths to decrease hydrogen ion concentration and to improve the surface finish obtained. As will be shown later, these additions to the standard solution appear to have little influence on the embrittling characteristics of the bath.

Bend Test Procedure

The test technique employed in this investigation is a free end-loaded bend test performed at a variety of fixed bending speeds. The test is shown schematically in figure 2 and is simply a slender column loaded in compression as one would squeeze a long thin sample between the jaws of a vise. Photographs of the actual bend test maching are shown in figures 3 and 4. The distance the jaws come together before a crack is observed or the sample completely fractures, whichever occurs first, was used as a measure of hydrogen embrittlement when compared to unembrittled base metal behavior under the same testing conditions. The comparison must be performed on materials having the same thickness in order to be completely valid.

When the sample fractured completely and fell out of the jaws, the machine stopped and the head depression was recorded automatically on a counter (see figures 3 and 4). If cracking did not result in complete fracture and the sample remained in the jaws the head depression at the appearance of the first crack was determined from the load verses time curve recorded automatically through a load ring-amplifier-recorder system (see figure 4).

Tests were carried out at four different testing speeds since detection of hydrogen embrittlement is known to be strain rate sensitive. Jaw closure rate varied in four steps: 0.001 in/min., 0.01 in/min., 0.1 in/min. and 1.0 in/min. The maximum testing time (for 2.8 inch Jaw closure) did not exceed 47 hours at 0.001 in/min., 5 hours at 0.01 in/min., ½ hour at 0.1 in/min. and 3 minutes at 1.0 in/min. These times correspond to the maximum jaw movement of the machine.

To compare the effects of various treatments on materials, a base line condition was established. In this investigation the bend ductility of the as-received or as-heat treated condition, depending on the material, was established as the base line. To be completely valid all further comparisons must be made to this base line using the same thickness of material. In order to investigate the effect of chemical milling, then, material was provided with thicknesses greater than the thickness of the base line material and were then chemically milled to the thickness of the base line material.

To accomplish this in the present investigation several gauges of each material were obtained, all from the same heat. For example, in the case of 4340 steel sheets of .040, .050 and .060 gauge material all rolled from the same heat were purchased from a supplier. In some other cases it was not possible to secure various gauges from one heat. Under these circumstances a heavy gauge material was obtained and the thinner material required to establish the unembrittled base condition was obtained by surface grinding to the desired gauge.

In general, five samples were run at each strain rate. Five samples gave a good indication of reproducibility of data and provided a sound basis for comparison of data. Test were started immediately after chemical milling (within five minutes) or after sime fixed recovery treatment. In general, the recovery treatment consisted of holding at room temperature for fixed periods of time. In a few cases elevated temperature recovery treatments were employed.

EXPERIMENTAL RESULTS AND DISCUSSION

Steel Alloys

AISI 4340 Steel

AISI 4340 steel heat treated to 265,000 psi ultimate tensile strength was chemically milled from .060" gauge and 0.050" gauge to 0.040" gauge. Time in the milling bath was about 25 minutes for .020" metal removal and 10 minutes for 0.010" metal removal. The average values of the bend ductility of the steel tested immediately after chemical milling verses testing speed are shown in figure 5. Data are tabulated in Table 3. As shown in figure 5, both 10 and 20 mils metal removal results in hydrogen embrittlement of the steel. More severe embrittlement results from the removal of 20 mils metal (longer time in bath). However, the effect of strain rate appears just opposite to what is normally found in hydrogen embrittled steels; i.e., according to these data embrittlement is more severe at high strain rates and completely dissappears at low strain rates. This is similar to the high strain rate embrittlement caused by hydride precipitation in some titanium alloys (30,31). However, precipatation of a hydride is not a reasonable explanation to account for the observed phenomenon in steel.

A second, more likely explanation can be found in the high diffusion rate of hydrogen in steel at ambient temperature. It appears possible that enough hydrogen will diffuse out of the steel during the long time (33 hours) involved in the low speed test that embrittlement will be completely eliminated. If this explanation is valid, ambient temperature recovery treatments should show a decrease in the degree of embrittlement with increased holding time before test. Furthermore, based on the data of figure 5, a 4 hour delay should not completely eliminate embrittlement but longer times should.

To investigate this further, a series of delay tests were performed on 4340 steel chemically milled to remove 20 mils of metal. Delay periods of 4 hours and 8 hours were selected. The results are shown in figure 6 and data tabulated in Table 4.

As shown in figure 6, the degree of embrittlement decreased with increased holding time at ambient temperature. Embrittlement was not completely eliminated with the 4 hour delay at room temperature but certainly has decreased by a considerable margin. After 8 hours at ambient temperature embrittlement appears to have decreased a little further. Longer delay tests were not carried out, but it is obvious from the data that recovery of ductility is at first rapid (4 hours caused a marked increase in ductility) but then slows down considerably (8 hours delay did not produce a large change over the 4 hour delay). It appears that a delay period longer than 8 hours but shorter than 33 hours is required for complete restoration of bend ductility. It is possible that the strain gradient established in the bend specimen during a test promotes the diffusion of hydrogen out of the steel, and, if this is true, longer delay periods or elevated temperature recovery treatments may be required to completely eliminate hydrogen embrittlement in unstressed specimens.

Thus, it is clear that hydrogen diffuses out of the chemically milled bend samples during the bend test, and therefore explains the apparent strain rate anomoly.

Cold Rolled 301 Stainless Steel

Stainless steel, type 301, cold rolled to 210,000 psi ultimate strength was chemically milled from 0.100 gauge to 0.060 gauge and tested for hydrogen embrittlement either immediately after chemically milling or after several different recovery treatments. Data are plotted in figure 7 and tabulated in Table 5.

As can be seen, 301 stainless steel cold-rolled to high strength is severely embrittled by hydrogen. At first this may seem contrary to expectations since 301 stainless steel is known as an austenitic stainless steel. Although 301 is austenitic in the annealed condition, when this alloy is severely cold-worked the relatively unstable austenite largely transforms to a low carbon martensite. Thus, cold worked 301 stainless steel would be more prone to hydrogen embrittlement than annealed 301 stainless steel and in view of this the present results are not unexpected.

Bend ductility on the same material chemically milled in a proprietary commercial bath are presented in figure 7 for comparison. In the one case, where the chemical milling bath was quite old, embrittlement established by the commercial bath was more severe than that found in samples chemical milled in the simple formulation used in the laboratory. But chemical milling in the new proprietory commercial bath (same formulation) resulted in somewhat less severe embrittlement. However, all chemical milling baths resulted in fairly severe embrittlement of the cold rolled 301 stainless steel.

It is to be noted that the strain rate sensitivity of the hydrogen embrittlement in this material is the same as found by most investigators for steel alloys; i.e. embrittlement is more severe at low strain rates. Comparing these results to those of 4340 steel (compare fig. 5 to fig. 7) strongly indicate that hydrogen is retained in the severely cold worked stainless steel at room temperature, whereas it diffused rapidly out of the 4340 steel. Consequently, ambient temperature recovery treatments would not be expected to provide much relief from embrittlement.

Tests at 0.1 in/min testing speed were run on samples chemically milled and held as long as 260 hours at room temperature. The results (see Table 5) were somewhat erratic but showed that little or no benefit was derived from this recovery treatment. High temperature recovery treatments (baking) were employed in an attempt to eliminate embrittlement. The results are shown in figure 7 and tabulated in Table 5. As shown in figure 7, 4 hours at 400°F reduced hydrogen embrittlement considerably but did not completely eliminate it. Longer baking at 400°F (8 hours) and baking 4.5 hours at 500°F further decreased embrittlement but again did not completely eliminate it. Although the bend ductility determined in the latter two tests is similar to the base metal ductility there was no doubt that some embrittlement remained. The base metal bend ductility maximum was limited by the bend test machine; i.e. samples were bent to the maximum of the machine at all testing speeds without cracking. The baked samples were always found to crack even if the head depression valve (bend ductility) was similar to the base metal value. However, the degree of embrittlement associated with this cracking is quite small.

It is reasonable to expect that higher temperature or longer time baking treatments will completely restore ductility. If such treatments are undesirable vacuum degassing at a relatively low temperature should be adequate.

Thus, 301 stainless steel cold worked to high strength levels can be severely embrittled by chemical milling. Furthermore, the embrittlement is difficult to eliminate in comparison to the 4340 steel. Baking treatments similar to those applied to cadinum plated steel are not completely adequate. Higher temperature baking treatments might suffice but were not investigated in the present study. Furthermore, higher baking temperatures may cause undesirable changes in the material, such as carbide precipitation in slip planes or grain boundaries or softening of the material by annealing the strained austenite.

The tensile properties and hardness of the cold worked 301 stainless steel are considerably below those of the heat treated 4340 steel (210 Ksi Ultimate and 42 R_C hardness compared to 265 Ksi ultimate and 52R_C hardness) and the carbon content of the martensite formed in each differ markedly. Based only on these comparisons 4340 steel should be subject to much more severe embrittlement than the cold worked 301 stainless steel. However, the degree of embrittlement appears to be similar for both materials. Obviously, other factors are involved. An obvious effect is that of cold work, per se. Zapffe and Haslem (19) have shown that cold worked low alloy steel is more susceptible to hydrogen embrittlement than annealed steel and, Darken and Smith (18) have shown that cold worked low alloy steel absorbes more hydrogen than annealed steel. Consequently, it is reasonable to attribute at least some part of the susceptibility of the cold worked 301 stainless steel to hydrogen embrittlement to cold work, as-such.

Other Steels

The remaining steels, heat treated 431 stainless, A286 stainless and AM350 and cold worked AM 355, were chemically milled to obtain between 20 and 25 mils metal removal and were then checked for hydrogen embrittlement. In all cases it was found that no embrittlement occured. In both the embrittled as well as the unembrittled condition all samples were bent to the limit of the bend test machine without fracture. Data are presented in Table 6.

These results are somewhat surprising. Although it was antisipated that the low strength austenitic A286 alloy would not be embrittled, it was expected that the other alloys certainly would be subject to hydrogen embrittlement. The ultimate tensile strength level of the other alloys varied from 195 Ksi for 431 stainless steel to 277 Ksi for the AM 355 cold rolled steel (Table 2). Furthermore, all three materials were hardened by the formation of martensite, a structure which is generally quite susceptible to hydrogen embrittlement. In addition, the AM 355 alloy was cold worked about 60 percent. Thus, all factors favorable to a high susceptibility to hydrogen embrittlement are present in these alloys.

Differences in chemical composition of these alloys may help explain this phenomenon. The major difference in chemical composition between 4340 steel and all other steels so far discussed is the increase of chromium from 0.80% in 4340 steel to about 15-17 percent in the other alloys. However the cold worked 301 stainless steel contains about 17% chromium but is still susceptible to hydrogen embrittlement. The difference between 301 composition and the composition of the other steels is the considerably higher nickel content and the absence of molybdenum.

These data suggest that with high chromium content ferritic or martensitic alloys resistance to hydrogen embrittlement is obtained by keeping nickel content low. However this correlation is far from precise.

The difference in hydrogen embrittlement susceptability may be related to composition through its effects on lattice parameter, hydrogen diffusion rate and/or hydrogen solubility although the actual mechanism is not known at present.

TITANIUM ALLOYS

Alpha Alloy Ti-5Al-2.5Sn

As received (mill annealed) alloy was chemically milled to remove 20 mils of material (.05 to .03) and tested for hydrogen embrittlement. All samples (both .030 gauge base metal and .030 gauge chem milled) bent to the full extent of the testing machine without failure indicating no hydrogen embrittlement susceptibility. Data are presented in Table 7.

Troiano, et al (29) have shown the Ti-5Al-2.5Sn alloy to be susceptable to hydrogen embrittlement in notched rupture tests at hydrogen levels greater than 300 ppm, but the range of stresses over which hydrogen - induced delayed failures occurred was not large because of the relatively slow diffusion rate of hydrogen in the alpha alloy.

In the present work, either the hydrogen level was insufficient to cause hydrogen embrittlement or the test was too rapid, even at the lowest testing speed, to detect its pressure.

Alpha-Beta Alloy Ti-6Al-4V

With 30 mils metal removal by chemical milling the alpha plus beta alloy Ti-6Al-4V exhibited some tendency toward hydrogen embrittlement. Data are given in Table 8 and plotted in figure 8. A rather wide range of values was obtained from 5 identically chemically milled samples. The values ranged, at some testing speeds, from a very low bend ductility to completely ductile behavior. Thus, at some testing speeds the range of values overlapped the "unembrittled" base metal condition. In figure 8, the dashed lines connected by the cross hatching represents the maximum range of values when at least one of the values showed complete ductility (i.e. no fracture at limit of machine). The solid line connecting the square's in this figure represents the average bend ductility value of those chamically milled samples that fractured.

In spite of the large scatter of data, it is quite obvious that chemical milling does cause a reduction in bend ductility and this in turn is probably due to the presence of hydrogen.

It is difficult to determine the strain rate sensitivity of this embrittlement from the data presented in figure 8. Based on average values there appears to be both low strain rate and high strain rate embrittlement sensitivity.

As mentioned previously, low strain rate embrittlement has been associated with hydrogen in solution while high strain rate embrittlement is associated with the precipitation of a hydride. Consequently, it should be possible to find hydrides in the microstructure of the chemically milled Ti 6Al-4V alloy if the high strain rate sensitivity is real and not data scatter.

A careful examination of the microstructure of the chemically milled samples did not reveal hydride precipitation. However, this examination was carried out at optical magnifications and it is possible that an extremely fine hydride precipitate may have gone undetected. Electron microscopy was not attempted. The alpha-beta alloys have been the most extensively investigated titanium alloys in so far as hydrogen embrittlement is concerned. These alloys have been found sensitive to hydrogen at low strain rates. Of all the alloys investigated the 6Al-4V alloy was found to be one of the least sensitive to embrittlement from hydrogen (28, 30, 32). The results of the present investigation tend to confirm the previous work since hydrogen charging conditions were rather severe and only minor embrittlement was found.

Beta-Alloy Ti-13V-11Cr-3Al

The all beta alloy Ti-13V-11Cr-3Al was severely embrittled by hydrogen with the removal of 30 mils of metal by chemical milling. The bend ductility data are plotted in figure 9 and tabulated in Table 9. As shown in figure 9 bend ductility of the chemically milled Ti-13V-11Cr-3Al alloy drops to only a small fraction of the unembrittled condition. There is no obvious strain rate sensitivity in this severely embrittled condition, at least over the range covered in this work.

Recovery treatments as long as 100 hours at room temperature did not produce any real change in bend ductility. Vacuum recovery treatments of 24 hours at either 800°F or 1100°F at a pressure of 40 microns or less did not increase bend ductitity appreciably. The results of the recovery treatments suggest very strongly that the severe embrittlement of the Ti-13V-11Cr-3Al alloy is primarily caused by the formation of a fairly stable hydride and the hydrogen in solution is not very detramental.

Troiano, et al have shown that the all β alloy Ti-13V-11Cr-3Al is susceptible to low strain rate hydrogen embrittlement attributable to hydrogen in solution at hydrogen levels above 400 ppm (29). Despite the high diffusion rates of hydrogen in beta alloys, the low strain embrittlement was not as pronounced as in alpha-beta alloys, probably because of the increased solubility or tolerance for hydrogen in the beta structure. Since low strain rate embrittlement due to hydrogen in solution was not found in the present study it may be concluded that either the test technique is not sensitive enough or that the severe embrittlement apparently caused by the hydride formation simply marked the low strain rate embrittlement.

Examination of the microstructure of the chemically milled Ti-13V-11Cr-3Al alloy failed to reveal any hydride precipitation. The microstructure "as heat treated" and "as chemically milled" is shown in figure 10 at 1000 X. The microstructure of this heat is somewhat unusual. Aging response was quite erratic. After the aging heat treatment, precipitation was complete in about 75 percent of the grains but 25 percent of the grains showed only minor response to the aging treatment and remained essentially unchanged (i.e. partially transformed Beta), see Figure 10.

However, no discernable or obvious hydride precipitation was apparent. Jaffer, Schwartzberg and Williams found impact or high strain rate embrittlement in a Ti-20 Mo beta alloy (28). They could not find hydride precipitation in any of the embrittled samples.

Low temperature vacuum recovery treatments should restore ductility if hydrogen in solution is the cause of embrittlement, but if the hydrogen is tied up in a more stable configuration high temperature vacuum treatments should be required.

Low temperature vacuum treatments of 24 hours at 800°F and 1100°F did not restore ductility (see Table 9). However, a vacuum degassing treatment of 24 hours at 1400°F was successful (see Table 9) but this treatment also resulted in resolution of the precipitate and considerably softened the alloy. Even though the base or comparison condition was changed by this treatment, the results are clear evidence of the removal of hydrogen from Ti-13V-11Cr-3Al alloy.

These data strongly suggest that hydrogen in a relatively stable form is the cause of embrittlement. It is possible that very fine hydrides are formed in the grain boundries but have gone undetected in the present investigation.

CONCLUSIONS

The processing technique of chemical milling can introduce hydrogen into most metals. The presence of the hydrogen may cause hydrogen embrittlement.

Whether a metal becomes hydrogen embrittled from the chemical milling process depends on many factors, all of which are not well defined or understood. Major factors such as chemical composition, structure and ultimate tensile strength are important, but are not the only factors involved. For example, in the present work, high strength 4340 steel and cold worked 301 stainless steel were found to be hydrogen embrittled from the chemical milling process, but other steels such as 431 stainless, AM 350 and AM 355 having similar composition, structure and tensile strength were not. The low strength austenitic stainless steel, A 286, was not hydrogen embrittled by chemical milling.

Of the three titanium alloys, the beta alloy, Ti-13V-11Cr-3Al was most severely hydrogen embrittled by chemical milling. The Alpha-beta alloy, Ti-6Al-4V, showed some minor embrittlement while the alpha alloy, Ti-5.0Al-2.5Sn was not embrittled.

Recovery treatments were found which restored the original ductility to the hydrogen embrittled materials. The ease of restoration of ductility (removal of hydrogen embrittlement) varied with the materials. Ductility was restored in the 4340 steel by holding between 8 and 33 hours at room temperature, but elevated temperature baking treatments were required for cold worked 301 stainless steel. Elevated temperature vacuum treatments were necessary in order to restore ductility to the Ti-13V-11Cr-3Al titanium alloy.

It is possible to use chemical composition, structure, and ultimate tensile strength as a guide in predicting the susceptibility of a given material to hydrogen embrittlement, but as shown by the present work this approach is far from precise. It appears necessary to check each material by a suitable test technique.

The constant strain rate bend test has proved a valuable testing tool in the evaluation of susceptibility of materials to hydrogen embrittlement. It is inexpensive of time and money.

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TABLE I

CHEMICAL COMPOSITION OF MATERIALS

COMPOSITION	4340	431	AM350	301	AM355	A286	Ti-5.0 Al-25 Sn	Ti-6Al-4V	Ti-13V-11Cr-3Al
C	0.41	0.20 Max.	0.14	0.12	0.13	0.065	0.15 Max.	0.10 Max.	0.05 Max.
Mn	0.67	-	0.75	1.12	0.81	1.25	0.3 Max.	-	-
P	0.008	-	0.015	0.018	0.016	0.025	-	-	-
S	0.018	-	0.019	0.010	0.011	0.013	-	-	-
Si	0.27	-	0.30	0.36	-	0.82	-	-	-
Cr	0.80	15 - 17.0	15.50	17.34	15.50	15.04	-	-	10-12
Mo	1.82	-	2.83	-	2.78	1.25	-	-	-
Ni	-	1.25 -2.50	4.34	7.10	4.27	25.73	-	-	-
N	-	-	0.12	0.024	0.11	-	0.07 Max.	0.05 Max.	0.08 Max.
Al	-	-	-	-	-	0.23	4.0 - 6.0	5.5 - 6.75	2.5 - 3.5
V	-	-	-	-	-	0.26	-	3.5 - 4.5	12.5 -14.5
Ti	-	-	-	-	-	2.12	Rem.	Rem.	Rem.
Fe	Rem.	Rem.	Rem.	Rem.	Rem.	Rem.	0.5 Max.	-	-
O_2	-	-	-	-	-	-	0.20 Max.	0.15 Max.	0.20 Max.
H_2	-	-	-	-	-	-	.003- 0.02	0.015 Max.	0.02 Max.
Sn	-	-	-	-	-	-	2.0 - 3.0	-	-
						0.005B			

TABLE 2

THERMAL TREATMENT AND SOME MECHANICAL PROPERTIES OF MATERIALS

MATERIAL	THERMAL TREATMENT	F_{tu}	F_{ty}	HARDNESS Rc
4340	1525°F for 30 minutes, O.Q. + 4 hours at 400°F, A.C.	265	229	52
431	1900°F for 30 minutes, O.Q. + -100°F for 30 minutes + 3 hours at 400°F	195[1]	-	40
AM350	1710°F for 30 minutes, W.Q. + -100°F for 3 hours + 850°F for 3 hours	200	160	45
301	As Received- Extra full hard	210	179	42
AM355	As Received- CRT	277	260	54
A286	1800°F for 30 minutes, A.C. + 16 hours at 1100°F, A.C.	128	62	20
Ti-5Al-2.5Sn	As Received- Annealed	115[2]	110[2]	36
Ti-6Al-4V	1725°F for 30 minutes, W.Q. + 2 hours at 1050°F, A.C.	160[2]	150[2]	36
Ti-13V-11Cr-3Al	900°F for 72 hours	190[2]	170[2]	39

1. Estimated from hardness
2. Guaranteed minimum values

TABLE 3

BEND DUCTILITY OF AISI 4340 STEEL

MATERIAL CONDITION		TESTING SPEED, IN/MIN. 1.0	0.1	0.01	0.001
AS HEAT TREATED- .040		2.45	2.27	2.21	2.04
		2.23	2.20	2.09	1.98
		2.36	2.35	2.13	2.04
		2.41	2.30	2.02	1.99
		2.30	2.34	2.15	2.05
	AVERAGE	2.35	2.30	2.12	2.01
HEAT TREATED + CHEMICALLY MILLED FROM .050 to .040, TESTED IMMEDIATELY		2.09	2.05	1.98	1.97
		2.19	2.09	1.93	2.03
		2.24	2.17	1.98	2.10
		2.16	1.81	1.96	2.01
		2.30	2.12	1.98	1.96
	AVERAGE	2.20	2.05	1.83	2.01
HEAT TREATED + CHEMICALLY MILLED FROM .060 to .040, TESTED IMMEDIATELY		1.42	1.58	1.72	2.09
		1.68	1.40	1.89	2.02
		1.40	1.60	1.45	2.07
		1.57	1.32	1.91	2.10
		1.86	1.50	1.92	2.09
	AVERAGE	1.59	1.48	1.78	2.07

TABLE 4

BEND DUCTILITY OF AISI 4340 STEEL

Recovery Treatments

MATERIAL CONDITION		TESTING SPEED, IN/MIN.			
		1.0	0.1	0.01	0.001
HEAT TREATED + CHEMICALLY MILLED FROM .060" to .040", HELD 4 HOURS AT AMBIENT TEMPERATURE BEFORE TESTING		2.45	1.98	2.16	-
		2.28	2.02	2.10	-
		2.22	2.01	2.07	-
		2.29	2.12	2.11	-
		2.30	2.01	2.03	-
	AVERAGE	2.27	2.03	2.10	-
HEAT TREATED + CHEMICALLY MILLED FROM 0.060" to 0.040", HELD 8 HOURS BEFORE TESTING		-	2.03	-	-
		-	2.12	-	-
		-	2.09	-	-
		-	2.04	-	-
		-	2.07	-	-
	AVERAGE	-	2.05	-	-

TABLE 5

BEND DUCTILITY OF COLD-ROLLED 301 STAINLESS STEEL

MATERIAL CONDITION		TESTING SPEED, IN/MIN. 1.0	0.1	0.01	0.001
AS COLD-ROLLED - 0.060 GAUGE		a	a	a	a
	AVERAGE	a	a	a	a
AS COLD-ROLLED - CHEMICALLY MILLED FROM .0.100 to 0.060, TESTED IMMEDIATELY		2.12	1.84	1.60	1.08
		2.36	1.99	1.58	1.28
		2.18	2.07	1.99	1.05
		2.36	1.96	1.67	1.10
		2.29	2.36	1.51	1.16
	AVERAGE	2.26	2.04	1.67	1.13
AS COLD-ROLLED - CHEMICALLY MILLED BY COMMERCIAL SOURCE- OLD BATH (.100 to 0.060) TESTED IMMEDIATELY		-	2.06	1.38	-
		-	1.88	1.48	-
		-	1.94	1.40	-
		-	2.04	1.46	-
		-	1.98	1.42	-
	AVERAGE		1.96	1.43	
AS COLD-ROLLED - CHEMICALLY MILLED BY COMMERCIAL SOURCE-NEW BATH, TESTED IMMEDIATELY		-	-	1.92	-
		-	-	1.61	-
		-	-	1.80	-
		-	-	1.70	-
		-	-	1.78	-
	AVERAGE			1.76	

TABLE 5 (Continued)

RECOVERY TESTS

TREATMENT	TIME, HOURS	BEND DUCTILITY AT 0.1 IN/MIN. TESTING SPEED
HELD AT AMBIENT TEMPERATURE	24	2.42, 1.56, 2.32, 2.20, 2.00 = 2.10
	48	2.20, 2.28, 2.36 = 2.28
	120	2.50, 2.30, 2.41 = 2.40
	260	2.05, 2.50, 2.45 = 2.33

HELD AT ELEVATED TEMPERATURES	TEMPERATURE °F	TIME, HOURS	BEND DUCTILITY AT 0.001 IN/MIN. TESTING SPEED
	400	2	<2.50[b]
	400	4	<2.50[b], 1.85, <2.50[b]
	400	8	2.6[c], 2.58[c]
	500	4.5	2.6[c], 2.62[c]

HELD AT 400°F	TIME	BEND DUCTILITY AT 1.0, 0.1 AND 0.01 IN/MIN. TESTING SPEED
	4	a

a. Samples bent to limit of machine, 2.67", without appearance of crack.
b. Value could not be accurately determined due to recorder failure.
c. Small cracks appear. Complete fracture did not take place.

TABLE 6

BEND DUCTILITY OF OTHER STEEL ALLOYS

MATERIAL	TREATMENT	BEND DUCTILITY AT ALL FOUR TESTING SPEEDS
A286 Stainless Steel	As Heat Treated (.040)	a
A286 Stainless Steel	Chemically milled from 060 to 040, tested immediately	a
431 Stainless Steel	As Heat Treated (.040)	a
431 Stainless Steel	Chemically milled from 060 to 040, tested immediately	a
AM350	As Heat Treated (.040)	a
AM350	Chemically milled from .063" to .040", tested immediately	a
AM355	As Received, (cold-rolled) .040"	a
AM355	Chemically milled from .063" to .040", tested immediately	a

a. All samples were bent to the limit of the bend test machine (2.67") without failure or cracking.

TABLE 7

BEND DUCTILITY OF THE ALPHA TITANIUM ALLOY, Ti-5Al-2.5Sn

MATERIAL CONDITION		TESTING SPEED, IN/MIN.			
		1.0	0.1	0.01	0.001
AS HEAT TREATED, .030" GAUGE		a	a	a	a
	AVERAGE	a	a	a	a
CHEMICALLY MILLED FROM .050" TO .030", TESTED IMMEDIATELY		a	a	a	a
	AVERAGE	a	a	a	a

a. All samples were bent to the limit of the bend test machine (2.67") without failure or cracking.

TABLE 8

BEND DUCTILITY OF THE ALPHA-BETA TITANIUM ALLOY Ti-6Al-4V

MATERIAL CONDITION		TESTING SPEED, IN/MIN.			
		1.0	0.1	0.01	0.001
AS HEAT TREATED, .040 GAUGE		2.57	2.65	2.65	a
		2.72	2.54	a	a
		2.58	2.65	a	a
		2.43	2.50	2.60	a
		2.43	2.61	a	a
	AVERAGE	2.55	2.59	a- 2.62	a
CHEMICALLY MILLED FROM .070" TO .040", TESTED IMMEDIATELY		2.15	a	a	1.99
		2.44	2.68	2.64	a
		2.75	a	2.35	1.65
		2.69	2.53	2.36	2.68
		2.06	a	2.69	a
	AVERAGE	2.42	a-2.60	a-2.51	a-2.11

a. All samples bent to the limit of the bend test machine (2.80") without fracture.

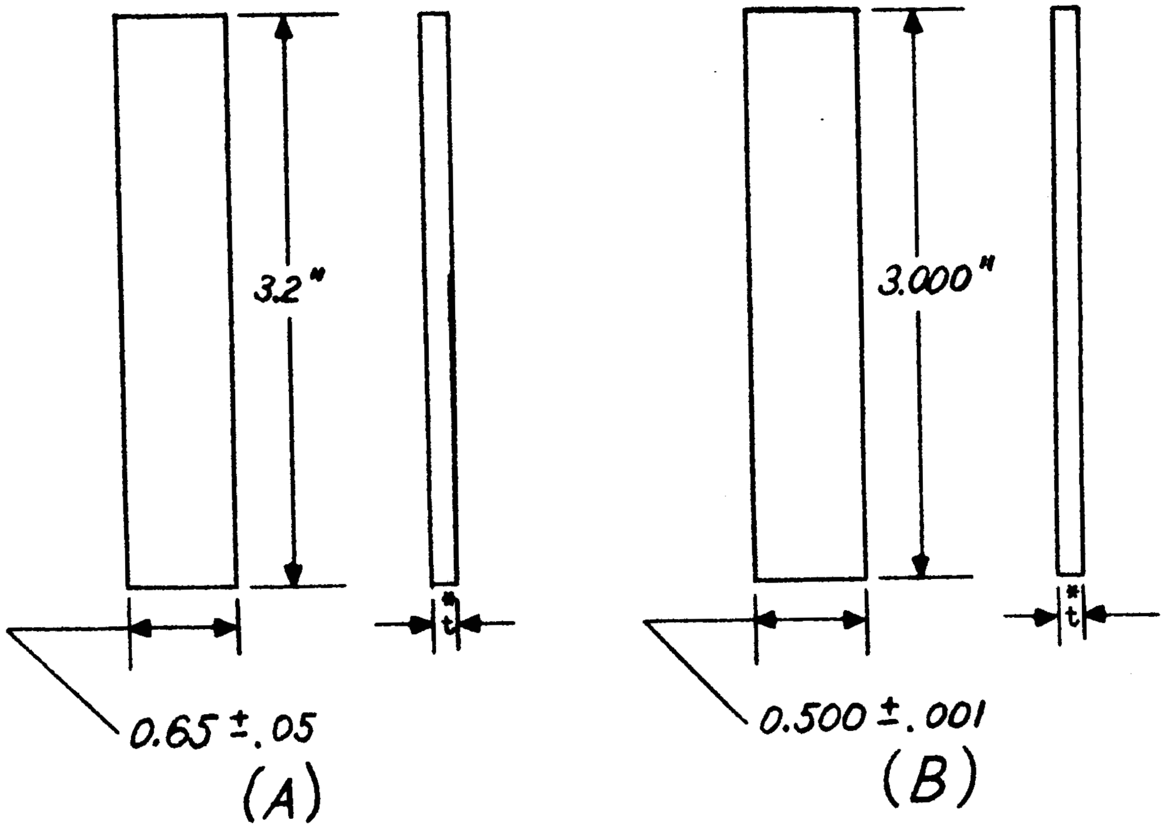
TABLE 9

BEND DUCTILITY OF THE BETA TITANIUM ALLOYS
Ti-13V-11Cr-3Al

MATERIAL CONDITION		TESTING SPEED, IN/MIN. 1.0	0.1	0.01	0.001
AS HEAT TREATED, 0.040 GAUGE		2.63	2.41	2.67	2.58
		2.70	2.51	2.62	2.39
		2.58	2.64	2.54	2.54
		2.50	2.52	2.65	2.50
		2.65	2.67	2.44	2.49
	AVERAGE	2.61	2.55	2.58	2.50
CHEMICALLY MILLED FROM 0.070" TO 0.040", TESTED IMMEDIATELY		0.32	0.30	0.52	0.20
		0.62	0.16	0.32	0.41
		0.25	0.42	0.24	0.32
		0.24	0.22	0.30	0.38
		0.36	0.28	0.33	0.25
	AVERAGE	0.36	0.28	0.34	0.31

RECOVERY TESTS

TREATMENT	TIME, HOURS	BEND DUCTILITY AT 0.1 IN/MIN. TESTING SPEED	
HELD AT ROOM TEMPERATURE	4	0.48, 0.59, 0.15, 0.21, 0.32	= 0.35
	24	0.32, 0.25, 0.20	= 0.25
	100	0.11, 0.11, 0.10	= 0.11
HELD AT 800°F IN VACUUM	24	0.07, 0.08, 0.10	= 0.08
HELD AT 1100°F IN VACUUM	24	0.32, 0.15, 0.20	= 0.22
HELD AT 1400°F IN VACUUM	24	No Failures in 0.01 and 0.001 in/min. test	



Size of Sheared Specimen

Size of Finished Bend Specimen

* Sheet Thickness

FIGURE 1. Preparation of bend test sample. (A) - Sheared to size. (B) - Ground to size.

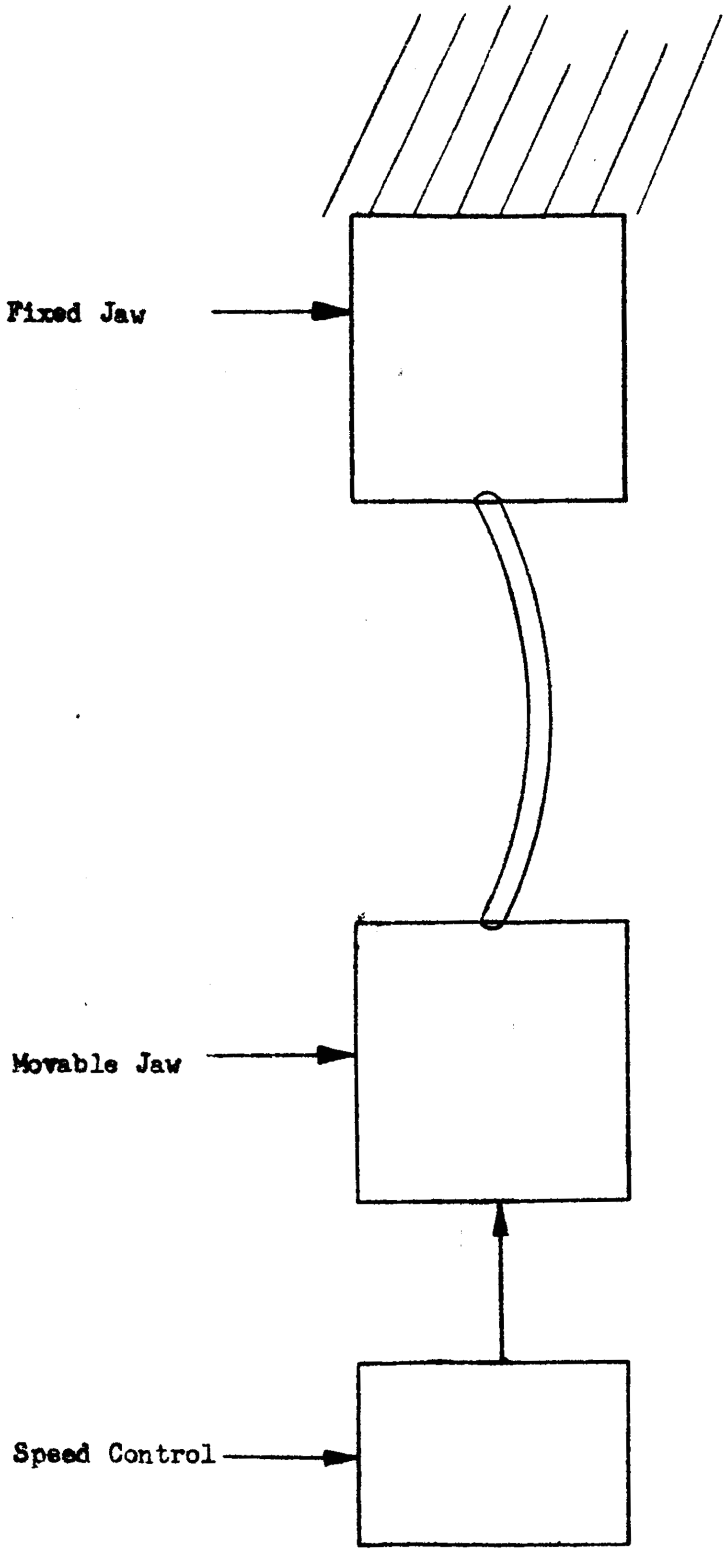


Figure 2. Schematic representation of free end-loaded bend test.

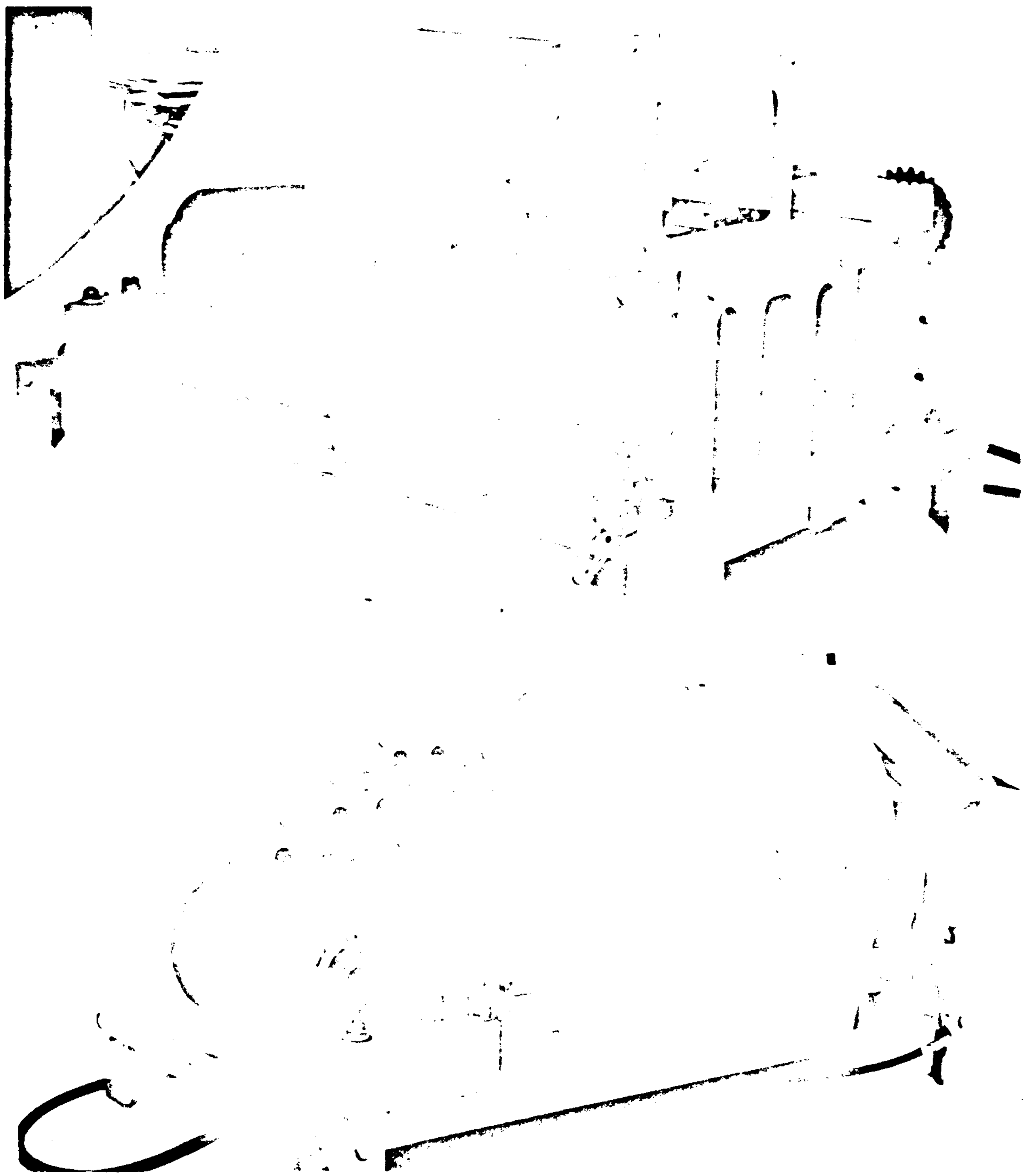


Figure 3. Bend Test Machine

Figure 4. Bend Test Machine with covers removed showing gear train, movable jaw, sample, and fixed jaw mounted on load ring.

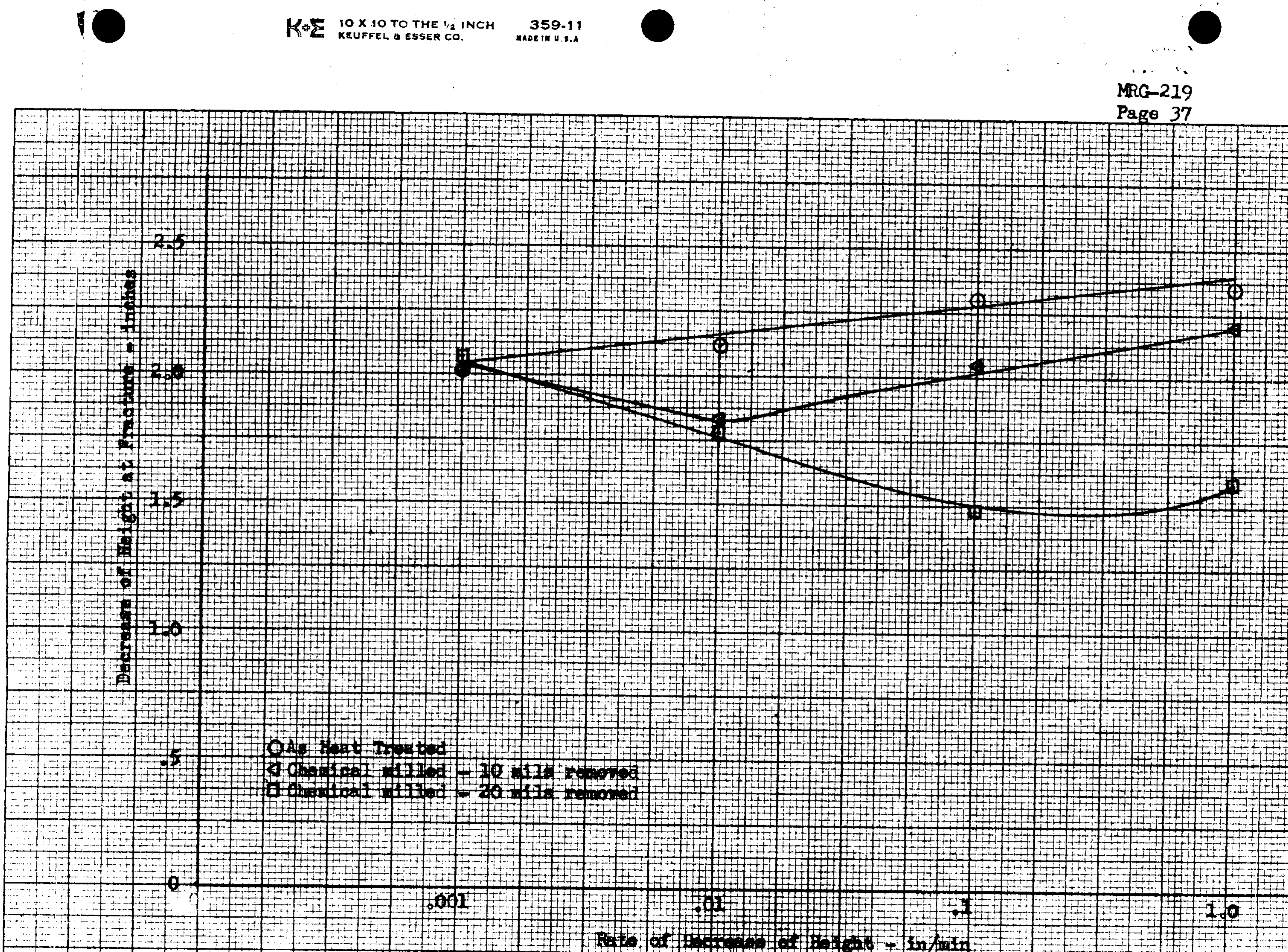


FIGURE 3. Effect of strain rate on the ductility of bend specimens of 4340. Tested immediately after chemical milling.

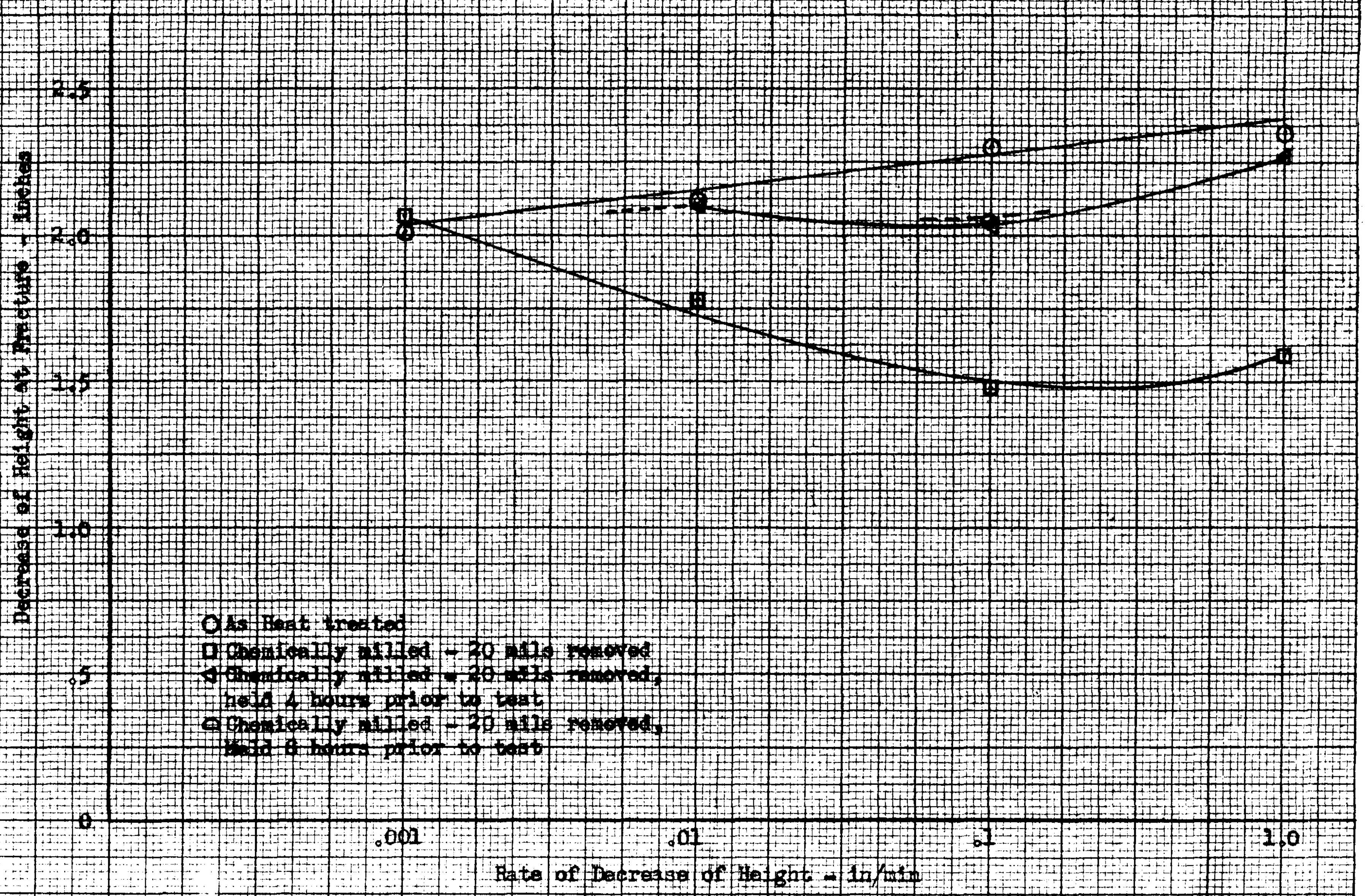


FIGURE 4. Effect of strain rate on the ductility of chemically milled bend specimens of AISI 4340 steel. Effect of recovery treatments at ambient temperatures.

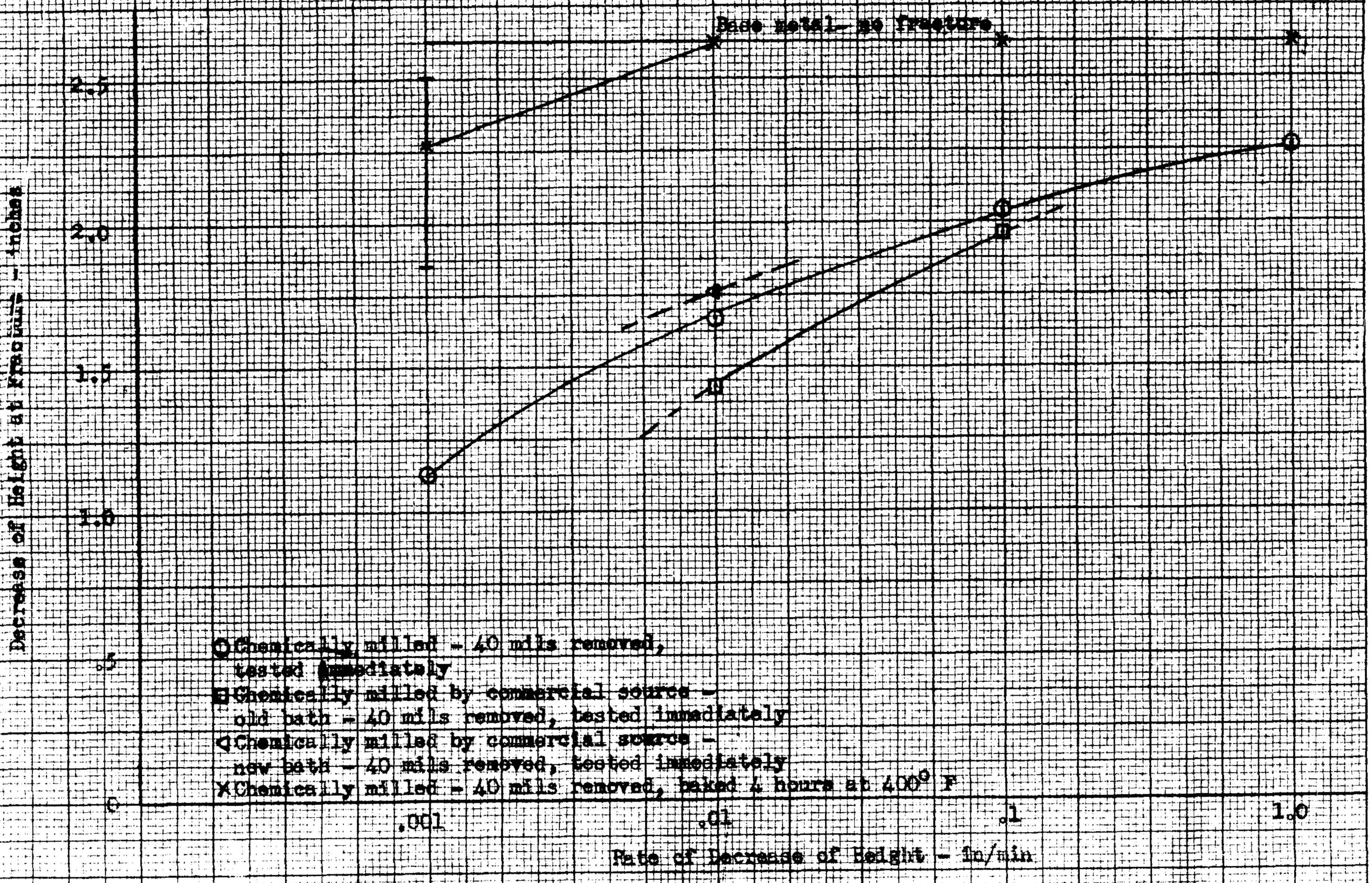


FIGURE 5. Effect of strain rate on the ductility of chemically milled bend specimens of cold-rolled 301 stainless steel.

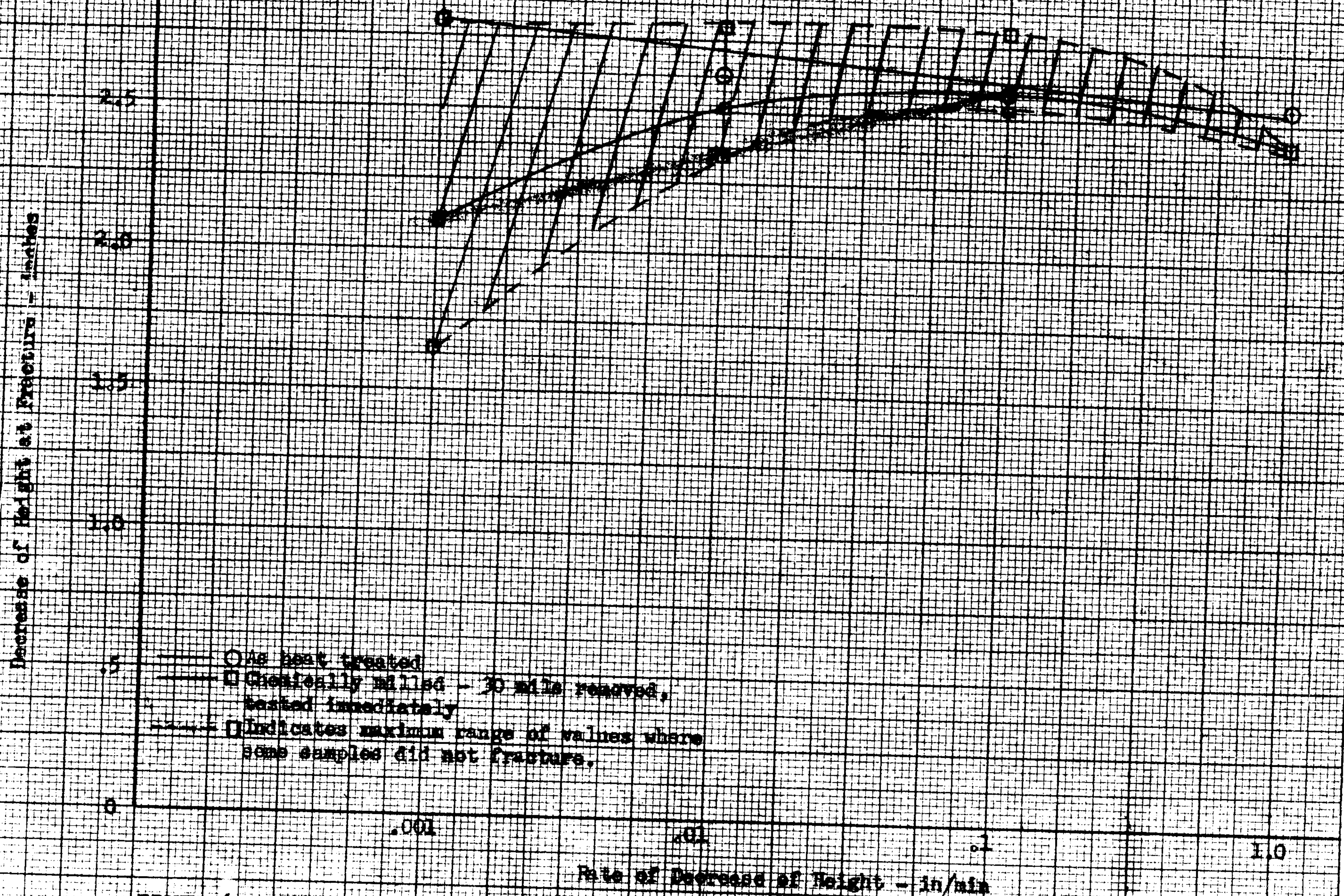


FIGURE 6. Effect of Strain rate on the ductility of chemically milled bend specimens of an alpha-beta Titanium alloy, Ti-6Al-4V.

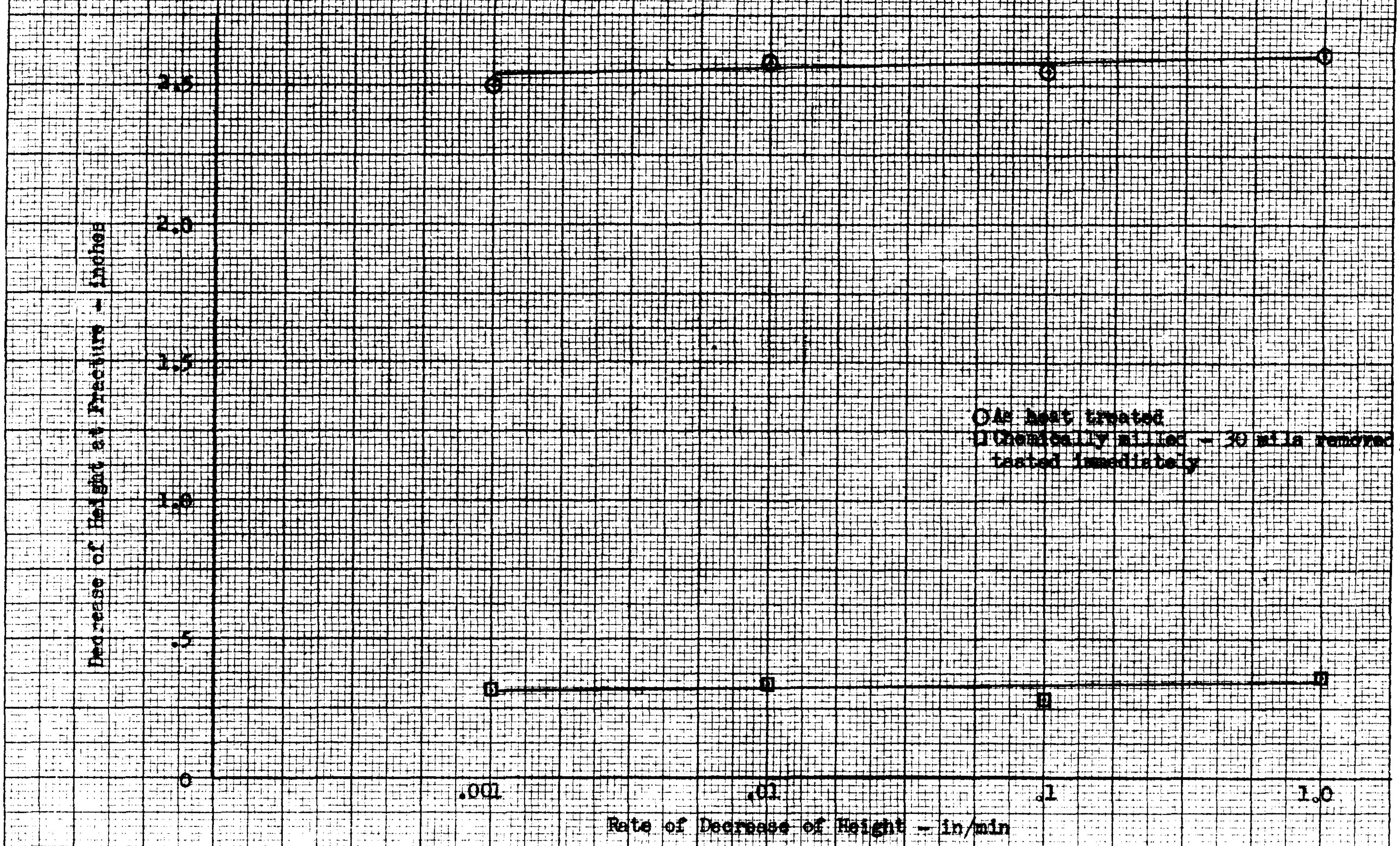


FIGURE 7. Effect of strain rate on the ductility of chemically milled bend specimens of a beta Titanium alloy, Ti-13V-11Cr-3Al.

M2765

As Heat Treated

M2766

Chemically Milled

Figure 10. Microstructure of Ti-13V-11Cr-3Al 1000 X